MISSOURI DEPARTMENT OF TRANSPORTATION
MATERIALS ENGINEERING
Jefferson City, Missouri

Test Method
MoDOT T70
GLASS BEAD TESTS

1.0 Scope.

This method covers the procedures for testing Type 1 and Type L reflectorizing glass beads for chemical resistance and embedment coating.

2.0 Procedures.

2.1 Water Resistance.

Ten ± 0.5 grams of beads placed in a Whatman single thickness cellulose extraction thimble, 33 by 80 mm, are refluxed for one hour in a Soxhlet extractor having an 85 mm siphon capacity using 150 ml of distilled water. All connections shall be ground glass. At the end of the refluxing period, allow the filtrate to cool to room temperature, and titrate with 0.1 normal hydrochloric acid, using phenolphthalein indicator. The beads shall be dried at 100 C, and examined for dulling under 60 power magnification.

2.2 Calcium Chloride Resistance.

Immerse approximately 10 grams of the beads in a 1.0 Molar calcium chloride solution for 3 hours. Rinse well, by decantation, with distilled water. Spread beads on a clean filter paper and allow to dry. Examine the beads for dulling under 60-power magnification.

2.3 Sodium Sulfide Resistance.

Immerse approximately 10 grams of the beads in a 50 percent solution of sodium sulfide for one hour. Rinse well, by decantation, with distilled water. Spread beads on a clean filter paper and allow to dry. Examine the beads for dulling under 60-power magnification.

2.4 Embedment Coating.

The embedment coating on Type L beads shall be tested in accordance with the following procedure.

2.4.1 Apparatus and Reagents.

Graduate Cylinder 50 ml
Glass Filter Paper 100 mm diameter
Dansyl Chloride - 98 percent
Scale - Analytical Balance (4 place)
Acetone - Reagent Grade
50 mm Buchner Funnel and Suction Flasks
Safety Glasses or Goggles
Darkened Glass Container (that can be sealed tightly)
Rubber Gloves (long sleeves)
Small Aluminum Weighing Dishes
Medicine Dropper
50 mm diameter Filter Paper (Whatman #1)
Vacuum Pump
Ultra-Violet Light Source - Intensity 7,000 uw/cm^2

Caution: Dansyl Chloride is a hazardous compound. Do not handle without protective gloves and safety glasses or goggles. Do not get onto skin.

2.4.2 Preparation of Dansyl Chloride Solution.

Prepare a solution by weighing 0.2 grams of Dansyl Chloride and dissolving it in 25 ml of acetone. This solution can be used for several tests during the day but must be kept refrigerated in a dark, tightly closed container between uses. Make a fresh solution daily.

2.4.3 Procedure.

(a) Set drying oven to 60 C. Turn on ultra-violet light.

(b) Weigh 2 samples of beads of 10 grams each. Place the sample to be evaluated in an aluminum weighing dish. Retain the other sample for a fluorescence observation comparison.

(c) Place a 50 mm diameter filter paper into the Buchner funnel and attach to the suction flask.

(d) Put the beads in the Buchner funnel and saturate the sample with the Dansyl Chloride solution using a medicine dropper. Let solution and sample stand for 30 seconds.

(e) Place the saturated beads into an aluminum dish and dry in oven 60 C at for 15 - 20 minutes. Beads will be yellow and agglomerated. Do not let the Dansyl Chloride solution char. (Properly discard the used filter paper because of the toxicity of the Dansyl Chloride.)

(f) Remove sample from the oven and place the glass beads in the Buchner funnel with new filter paper. Rinse the beads with 100 ml of Acetone. Use the suction during this step. All yellow must be removed from the beads.

(g) Remove the beads from the funnel and place into a new aluminum tray. Allow the beads to dry in the oven for 5 - 10 minutes until free flowing.

(h) Remove the beads from the oven and place on glass filter paper. If beads are agglomerated, break them up with a spatula.

(i) Inspect the treated sample under the ultra-violet light, in a darkened room.

2.4.4 Observations.

(a) Embedment coated beads will emit a yellow-green florescence.

(b) If additional fluorescence is observed when compared with the original untreated sample, the lot is accepted. If no additional fluorescence is observed, the test should be rerun using a new 10 gram sample of beads and a fresh solution of Dansyl Chloride.

(c) If no additional fluorescence is observed on the new sample of beads, the material is not properly coated and the lot is rejected. If additional fluorescence is observed, the sample is reported as treated with embedment coating material.